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**FORMULATION PROCEDURE AND SPECTRAL
DATA FOR A HIGHLY REFLECTING COATING
FROM 200nm TO 2300nm**

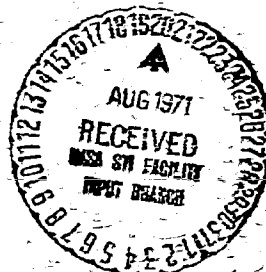
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ABSTRACT

A method for formulating a highly reflecting and mechanically stable coating from very pure barium sulphate is carefully detailed. Reflectance data are presented for this coating from 200 nm to 2500 nm and compared with the commercially available Eastman Kodak pre-formulated coating as well as freshly smoked magnesium oxide. Compared to the Eastman coating, the one detailed in the body of this report has the advantages of somewhat higher reflectance values throughout the spectral region of interest as well as greater ease of handling.

CONTENTS

	<u>Page</u>
ABSTRACT	iii
INTRODUCTION	1
FORMULATING PROCEDURE	2
SURFACE PREPARATION AND APPLICATION PROCEDURE	4
SPECTRAL REFLECTANCE DATA	5
Applications of MS-125	5
Future of Paints as Ultraviolet Reflecting Coatings	7

LIST OF ILLUSTRATIONS

<u>Figure</u>	<u>Page</u>
1 Comparisons of Spectral Reflectance Data for Mg O, MS-125, Eastman White Reflectance Standard and NaCl	6

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1 Formulation MS-125	3

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FORMULATION PROCEDURE AND SPECTRAL DATA FOR GSFC WHITE REFLECTANCE STANDARD

INTRODUCTION

Continuing interest in highly reflecting diffuse coatings has prompted investigations into the promise of such materials as the alkali metal halides, particularly sodium chloride;¹ the alkaline earth carbonates, particularly magnesium carbonate, aluminum oxide - magnesium oxide - potassium silicate paints, barium sulphate powder, barium sulphate powder in polyvinyl alcohol and smoked magnesium oxide to recall a few. Most often used among these candidates is smoked magnesium oxide. When freshly smoked, both its average diffuse reflectance and the magnitude of its reflectance from 200 nm to 2400 are unchallenged. This property is particularly true in the ultraviolet. However, the atmospheric instability^{2,3} and fragility of smoked magnesium oxide spurred investigations for the purpose of finding compromise coatings not suffering from the disadvantages of smoked magnesium oxide. This search is responsible for the list of materials and systems given above.

Although barium sulphate has been investigated in our laboratory previously, it was not until a high purity barium sulphate powder and a polyvinyl alcohol suspension of that powder were introduced by the Eastman Kodak Company that barium sulphate based diffuse reflectance coatings received extensive attention. A paper by Grum and Luckey⁴ summarizes the Kodak process for purification

¹Stuart, J. W., Tech. Brief 71-10110.

²Hammond III, H. K., J. Opt. Soc. Amer. 45, 904(1955).

³Weber, H., Z. Phys. 130, 392(1951).

⁴Grum, F., and Luckey, G. W., App. Optics, 7, 2289-2294(1968).

and precipitation of their high purity barium sulphate and gives the historical background serving to generate their interest in this particular powder. Also included in their publication is comparative data with smoked magnesium oxide. Because these authors are complete in their treatment of the virtues of barium sulphate powder and the polyvinyl alcohol coating formulation, we are content with meticulously treating the formulation problem. Experiences in our laboratory with the Eastman paint demonstrated that problems were usually encountered in its application because of phase agglomeration between pigment and pigment binder due to interactions. Redispersion of their formulation has been found difficult. It is with this aspect of the problem that this write-up is primarily concerned; namely, providing instructions for formulation of the coating wherefrom these aforementioned interactions are minimal. Also, the formulation as given is found after standing to be redispersable by hand stirring after the addition of water.

Included also are reflectance curves comparing both the Eastman coating and our coating with freshly smoked magnesium oxide. For easy reference, data on sodium chloride is also incorporated.

FORMULATING PROCEDURE

Referring to the formulation presented in Table I, the dominant vehicle, water, is split into two portions. The first portion, weighing 72 grams, is to be combined with the polyvinyl alcohol while the remaining portion is mixed with the absolute alcohol. After weighing out 72 grams of water into a suitable container, the water is heated to about 90°C or to a temperature sufficient for melting the polyvinyl alcohol. After the proper melting temperature for the polyvinyl

Table I
Formulation MS-125: Barium Sulphate Polyvinyl Alcohol Coating
With Extended White Reflectance

Ingredient	Weight (gms.)	Percent	Remarks
Barium Sulphate ($\text{Ba SO}_4 \cdot 2 \text{H}_2\text{O}$)	400	55.2	Eastman Kodak White Reflectance Standard No. 6091
Water	197	27.2	Distilled
Polyvinyl Alcohol	3	0.4	99% Hydrolyzed (Hot water soluble)
Ethyl Alcohol	125	17.2	Absolute

alcohol has been reached by the water, the polyvinyl alcohol solids are sprinkled slowly into the water, while the solids are kept under rapid agitation to prevent agglomeration and the formation of the PVA into large globules. Provided formulations of this coating are not excessive in size, this initial operation is most conveniently carried out using a magnetic stirrer. While the polyvinyl alcohol is dissolving — a procedure normally requiring about fifteen minutes — the remaining portion of water (125 grams) is mixed with the required 125 gram portion of absolute alcohol in a separate container.

Once the polyvinyl alcohol has achieved complete solvation, the solution is allowed to cool down to room temperature under continued agitation. Once room temperature has been reached by the solution, it is added to a blender jar. Subsequently the blender is operated at high speed after which the 250 grams of

alcohol-water solution is added slowly. Care must be taken to prevent dissolution of the polyvinyl alcohol. A safe duration time for this step is two minutes. Care must also be taken to prevent noticeable heating of the mixture.

Finally, the required portion of barium sulphate (400 grams) is added to the rapidly agitating mixture. The powder may be added rapidly. The required dispersion should be obtained in ten to fifteen minutes. Care must be taken once more to prevent over heating of the coating composition.

SURFACE PREPARATION AND APPLICATION PROCEDURE

Prior to application of the coating composition to metal surfaces, the surfaces must be thoroughly degreased. Since most applications of this coating are to small objects such as integrating spheres, degreasing may be readily accomplished by wiping with acetone, methyl ethyl ketone, etc. Only in unusual circumstances is alkali cleaning followed by acid etching required. Once the surfaces are grease free, a suitable prime coat must be applied. A satisfactory primer for this instance is a flat acrylic white. The intermediate application of a white primer is required because residual bisulphate or sulfuric acid moieties are gradually released from the surfaces of the pigment, which are capable of diffusing to the metal-coating interface and reacting with metal to form hydrogen. Simultaneously, sulfuric-bisulphate migration is paralleled by the counter diffusion of metallic particles and ions capable of staining or destroying the coating. Consequently, surface preparation and suitably precoat are important.

Prior to spray application, the coating composition as in the Formulating Procedure must be thoroughly stirred and diluted with sufficient distilled water

to allow the paint to regain its freshly formulated consistency. Normally after a shelf life of two weeks or more agglomeration can occur during spray application. Consequently, prior addition of water expedites application and provides the desired cosmetic appearance.

The coating composition as formulated is best applied using a spray apparatus incorporating a pressure pot. A suitable system is BINKS model number seven. As in the procedure recommended by Eastman Kodak, premarking the primed surface with a pencil provides a convenient way for monitoring the coverage of the coating over the primer. Normally this hide is achieved with about 40 mils of the barium sulphate coating.

After application, the coating is air-cured. Heating is unnecessary and is to be avoided.

SPECTRAL REFLECTANCE DATA

Spectral comparisons of the Eastman diffuse reflectance with the one described herein (MS-125) are presented in Figure 1 along with smoked Mg O and sodium chloride for the sake of completeness. Data for the Eastman, MS-125, and smoked Mg O coatings were obtained on a Cary model 90 spectrophotometer and normalized to absolute scale. Data for the sodium chloride coating were obtained by means of a Beckman PK-2A spectrophotometer with a Gier-Dunkle attachment.

Applications of MS-125

The use of barium sulphate dispersed in polyvinyl alcohol as an integrating coating is now nearly universal. Sodium chloride is also currently in use, but

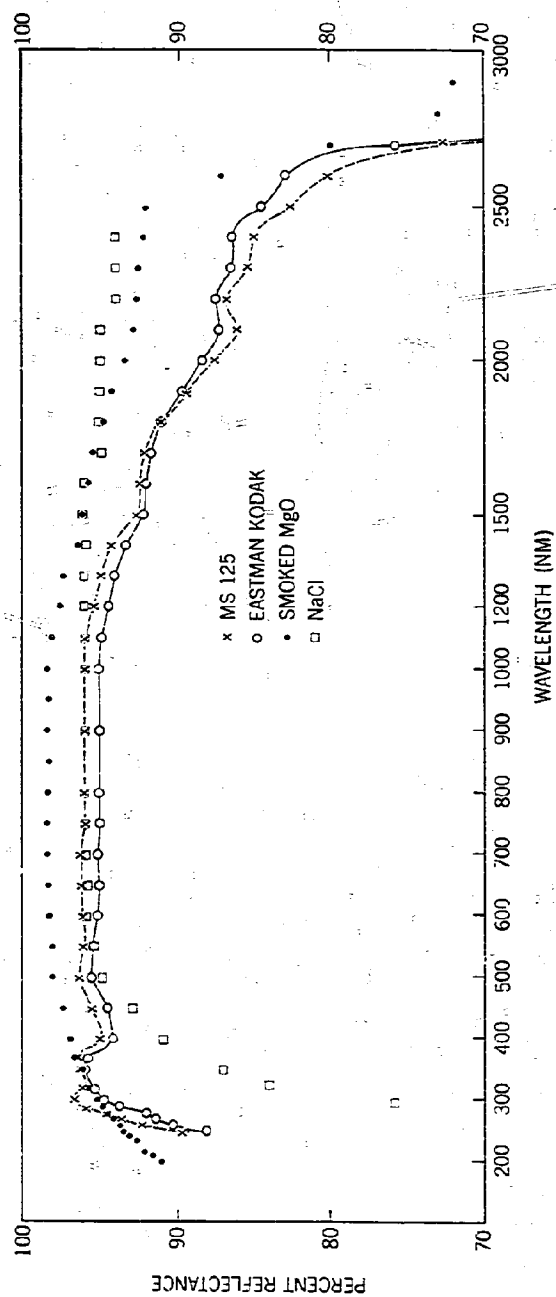


Figure 1. Comparisons of Spectral Reflectance Data for MgO, MS-125, Eastman White Reflectance Standard and NaCl

because application is more time consuming and requires considerable experience, its use lags MS-125. When the highest infrared reflectance is required, sodium chloride must be used; however, if high ultraviolet reflectance below 295 nm is required, use of MS-125 is required.

Diffuse reflecting white coatings are continually in demand for calibrating photomultiplier tubes used in flight experiments. Calibration coatings have been prepared for OAO-A, B, C and HEAO. Similarly, the Cerenkov chamber for HECRE has been coated with this composition because it currently offers the best reflectance in the neighborhood of 200 nm, as well as mechanical stability.

Uses for the Nimbus Project are numerous, but the continuing use is for coating one inch fused silica integrating spheres flown on sound rockets to obtain calibration points for the BACKSCATTER ULTRAVIOLET EXPERIMENT flown on D.

For ERTS A, a thirty inch aluminum sphere was coated, again to provide a suitable surface for calibration of a scanner with sensitivity in the ultraviolet, visible as well as infrared regions of the electromagnetic spectrum.

Future of Paints as Ultraviolet Reflecting Coatings

Within the context of the term ultraviolet reflecting as used in this section is meant high ultraviolet reflectance and diffuseness in the neighborhood of 200 nm. Smoked magnesium oxide not only exists as the most reflecting coating in this region but also the most diffuse.⁵ Optically therefore, barium sulphate and polyvinyl alcohol are less satisfactory. A major difficulty in further development of a

⁵Private communication from Arlen Kruger, Nimbus Project.

mechanically stable coating for this region is purity of the powder — not withstanding the fact that a binder could not be used since all are strongly absorbing in this region. The essential concepts required for a choice of powder are the ionization potential of the perspective cation and the electron affinity of the perspective anion. To maximize the photon energy for reflectance cut-off, the difference between these quantities must be maximized. From the periodic table therefore, cesium fluoride fits this condition best, but not optimally since it is too moisture sensitive. With elaborate containment precautions perhaps it could be used. Lithium fluoride is a more optimal choice than cesium fluoride because it is less moisture sensitive. Carrying on now with the subject of purity, it is clear that whenever a host like lithium fluoride, i.e., one with a wide band gap, is impure extraneous absorptions are introduced at the gap energies for the impurities because their ionization potential electron affinity differences are less than that of the host. It appears therefore that smoked magnesium oxide and barium sulphate-polyvinyl alcohol are optimal for their respective mechanical strengths.